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FRETTING OF  
NICKEL-CHROMIUM-ALUMINUM ALLOYS  
AT TEMPERATURES TO 816° C

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16. Abstract <p>A series of four nickel-based alloys containing 10 percent and 20 percent chromium in combination with 2 percent and 5 percent aluminum were fretted in dry air at temperatures to 816<sup>0</sup> C. At all temperatures, the alloys showed far less fretting wear than did high-purity nickel. This was attributed to the formation of protective oxide films on the alloys, the result of the selective oxidation of the alloy constituents. Increasing the aluminum concentration reduced fretting wear at all temperatures. Increasing the chromium concentration from 10 percent to 20 percent resulted in decreased fretting wear at 23<sup>0</sup> and 540<sup>0</sup> C, but increased fretting wear at 650<sup>0</sup> and 816<sup>0</sup> C.</p>					
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# FRETTING OF NICKEL-CHROMIUM-ALUMINUM ALLOYS AT TEMPERATURES TO 816<sup>0</sup> C

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## SUMMARY

Fretting experiments were conducted on 99.9-percent-pure nickel and on four nickel-based alloys containing 10 and 20 percent chromium in combination with 2 and 5 percent aluminum. Pairs of like-metal specimens were fretted at temperatures up to 816<sup>0</sup> C (1500<sup>0</sup> F) in dry air.

At all temperatures, the alloys showed far less fretting wear than did the 99.9-percent-pure nickel. X-ray dispersion analysis suggested that at elevated temperatures this lower fretting wear was due to the selective oxidation of alloy constituents in the fretting region. Superior resistance to fretting at high temperatures seemed to be associated with a preferential concentration of aluminum on the wear scar surface. This situation was observed in the 10-percent-chromium - 5-percent-aluminum alloy. In both 10- and 20-percent-chromium alloys, increasing the aluminum concentration reduced the fretting wear.

The effect of chromium additions was more complex than the effect of aluminum additions. Increasing the chromium concentration from 10 percent to 20 percent resulted in less fretting wear at 23<sup>0</sup> and 540<sup>0</sup> C, but greater fretting wear at 650<sup>0</sup> and 816<sup>0</sup> C.

## INTRODUCTION

The chromium in nickel- and cobalt-based superalloy systems significantly reduces the rate of high-temperature oxidation of the alloy by forming chromium-rich oxide films on the alloy surface (refs. 1 and 2). Earlier fretting work (refs. 3 and 4) has shown that nickel- and cobalt-based superalloy systems containing 15 and 20 weight percent chromium undergo much less fretting damage at high temperatures (up to 816<sup>0</sup> C) than they do at room temperature. This behavior has been attributed to the protective nature of

the chromium-rich oxide films and their ability to interfere with the adhesion between contacting metal surfaces.

At temperatures above  $950^{\circ}\text{C}$ , the chromium-rich oxide becomes unstable and a portion of it vaporizes (ref. 5), which leaves the surface unprotected. To extend the capability of nickel-based alloys to resist oxidation at elevated temperatures, various quantities, typically several atomic percent, of aluminum have been added. The effects that these aluminum additions might have on the fretting characteristics of the surface oxide films formed at temperatures up to  $816^{\circ}\text{C}$  are not known.

This investigation was conducted to determine the effect of varied aluminum and chromium concentration on the fretting of a series of nickel-chromium-aluminum alloys at temperatures to  $816^{\circ}\text{C}$ . The following are the compositions (by weight) of the alloys examined: nickel - 20 percent chromium - 5 percent aluminum; nickel - 20 percent chromium - 2 percent aluminum; nickel - 10 percent chromium - 5 percent aluminum; nickel - 10 percent chromium - 2 percent aluminum. Also, 99.9-percent-pure nickel was fretted at room temperature and at  $650^{\circ}\text{C}$ . All fretting was conducted in dry air (<100 ppm water) except for a series of tests on 99.9-percent nickel that was conducted not only in dry air, but also in 23 percent relative humidity, and in saturated air.

## APPARATUS

A schematic diagram of the fretting rig is shown in figure 1, and a detailed description of its operation is included in reference 6. In essence, a linear oscillatory motion is provided by an electromagnetically driven vibrator with the frequency controlled by a variable oscillator. The load is applied to the specimens by placing precision weights on a pan which is hung from the load arm.

The fretting specimens consist of an upper, stationary, 4.76-milimeter-radius, hemispherical tip in contact with a lower flat which is driven by the vibrator.

During high-temperature experiments, the specimens and grip assemblies were surrounded by a 310-stainless-steel susceptor which was heated by an induction coil. The temperature was monitored by a thermocouple probe mounted in the lower grip. The grips were specially designed so that they stored sufficient elastic energy to ensure that no slippage would occur as a result of differential thermal expansion.

A dry air environment was provided by flowing air through an absorption drier, then into the experimental chamber. In this way, moisture content was kept in the range of 10 to 100 parts per million. When a moisture-saturated environment was desired, the air was bubbled through a water-filled column, then led into the chamber.

## PROCEDURE

Before fretting, the flat specimens were lapped, mechanically polished with 0.05-micrometer alumina polishing compound, then rinsed in distilled water. The hemispherical tips, ground to a 0.1-micrometer ( $4\text{-}\mu\text{in.}$ ) finish, were scrubbed with levigated alumina, and rinsed.

Next, a pair of like-metal specimens was assembled into the experimental chamber, which was then purged with dry air (or saturated air, if desired) for 1/2 hour before fretting. For the elevated-temperature experiments, the specimens were brought to the desired experiment temperature during the purge by setting the plate current on the induction heater control so that the temperature stabilized at the desired level ( $504^{\circ}$ ,  $650^{\circ}$ , or  $816^{\circ}\text{C}$ ). With the vibrator now turned on, fretting was initiated by putting sufficient weight into the pan to just bring the specimens into contact (easily determined by a slight transmitted vibration). An additional 50-gram weight was then added to give a 1.47-newton normal load to the rubbing surfaces. Other experimental conditions included a peak-to-peak amplitude of 75 micrometers (0.003 in.), a fretting frequency of  $80\pm 0.2$  hertz, and a duration of 288 000 cycles (1 hr).

Just before the end of each fretting experiment, a friction measurement was made by recording the axial force in the shaft by means of a strain-gage ring located in the shaft between the driver and the experimental chamber. The normal load was then removed, ending the fretting process, and the force through the strain-gage ring was again recorded. The frictional force was the difference between the force measurements made with the normal load applied, and after the normal load was removed.

Following each fretting experiment, the fretting scar on the flat surface was photographed to record the size and features of the wear scar and the debris accumulation around the scar. The loose debris was then rinsed off with ethyl alcohol, and a light section microscope was used to measure the wear volume on the flat specimen.

Specimens that were examined in the scanning electron microscope (SEM) were ultrasonically cleaned in methanol before viewing, to remove as much debris still adhering to the wear scar as possible.

## MATERIALS

The metals examined in this study included 99.9-percent-pure nickel, and four nickel-chromium-aluminum alloys having the following compositions (by weight percent): nickel - 20 percent chromium - 5 percent aluminum; nickel - 10 percent chromium - 5 percent aluminum; nickel - 20 percent chromium - 2 percent aluminum; and nickel - 10 percent chromium - 2 percent aluminum. The alloys were prepared from elemental metals of 99.9+ percent purity, vacuum melted in a zirconium oxide ( $\text{Zr}_2\text{O}_3$ ) crucible

and cast in graphite molds. The 12- to 13-millimeter- (1/2-in. -) diameter cylindrical castings were then machined into specimens.

## RESULTS AND DISCUSSION

Table I shows the results of the friction measurements made on 99.9-percent-pure nickel and on the nickel-chromium-aluminum alloys. For the nickel and for all the alloys examined, the friction at elevated temperatures was higher than that at room temperature. At room temperature, the coefficient of friction was about 0.1 in all cases and it rose to about 0.3 to 0.4 at 816° C.

The coefficient-of-friction values of 0.1 measured at room temperature are quite low compared to values as high as 0.9 measured in unidirectional sliding experiments with nickel (ref. 7). This is because each friction measurement was made at the end of a fretting experiment, and the contact region was covered with oxidized debris which prevented direct metal-to-metal contact. For the first minute or two of a room-temperature fretting experiment, though, the coefficient of friction was considerably higher than 0.1 and very unsteady. This was indicated by large fluctuations in the strain-gage force and by variations in the vibration amplitude induced by instantaneous high friction. Generally, far less debris was generated during fretting at high temperature, as may be seen in figure 2, and the coefficient of friction values of 0.3 to 0.4 are similar to those measured during high-temperature, unidirectional sliding experiments (ref. 7).

The fretting wear volume measurements are shown in table II. The experiments performed on 99.9-percent-pure nickel at room temperature show an interesting wear volume dependence on relative humidity. The wear volume after fretting in dry air is three or four times as large as the wear volume after fretting in an environment with 25 percent relative humidity. However, fretting in saturated air produces only about 50 percent more wear than fretting under conditions of 25 percent relative humidity. Differences were observed in the darkness of the debris and the amounts of oxidized debris under different humidity conditions, as may be seen in figure 3. The area immediately surrounding the wear scar produced under saturated air conditions is covered with a very dark oxidized debris, which was not seen under the other humidity conditions.

The results of the experiments on nickel under varied humidity conditions suggest that two humidity-related effects are interacting. As the humidity is increased from the condition of dry air, the oxidized debris becomes less abrasive, and reduced fretting wear results. This effect has been proposed to explain the reduction in fretting wear with increased humidity in the case of carbon steel (ref. 8). As the humidity is further increased toward saturation, the slight increase in fretting wear may be attributed to

an increase in the rate of surface corrosion. It is interesting to note the strong effect that changing the relative humidity from nominally 0 to 25 percent has on the fretting wear of 99.9-percent-pure nickel.

Referring again to table II, wear measurements show that the alloy additions generally reduce fretting at all temperatures, with increasing effectiveness as the temperature increases. For all of the alloys studied, the fretting wear volume measured at 540° C was roughly an order of magnitude less than that observed at room temperature. Also, the fretting-wear volumes measured on the 20-percent-chromium alloys were less than those measured on the 10-percent-chromium alloys at room temperature and at 540° C. These observations are consistent with trends observed in reference 3.

At 650° C, the 20-percent-chromium alloys showed significantly more fretting wear than at 540° C. At 816° C, buildups of material (fig. 4(a)) were observed in the fretting scars of these alloys. On the other hand, the 10-percent-chromium alloys showed very little, if any, increased fretting wear as the temperature was increased from 540° C to 650° C; and the fretting-wear volumes measured on the 10-percent-chromium alloys were smaller than those measured on the 20-percent-chromium alloys. Also, no buildups of material were observed in the fretting wear scars of the 10-percent-chromium alloys at 816° C (fig. 4(b)).

At all temperatures, the 5-percent-aluminum alloys showed less fretting wear than did the 2-percent-aluminum alloys. Proportionally, the increase in fretting wear with increasing temperature observed at 650° C was about the same for the 20-percent-chromium - 5-percent-aluminum alloy and the 20-percent-chromium - 2-percent-aluminum alloy.

The trend of increasing fretting wear with increasing temperature above 540° C, especially in the 20-percent-chromium alloys, is contrary to the observations made on nickel- and cobalt-based alloys in reference 3. This is probably because the alloys studied in reference 3 were precipitation hardened and retained their hardness to temperatures of about 850° C. On the other hand, the nickel-chromium-aluminum alloys in this investigation were nonhardened alloys, subject to softening above about 600° C.

The observation that higher fretting wear occurred in the 20-percent-chromium alloys than in the 10-percent-chromium alloys at first seems difficult to understand in terms of protective oxide film formation. There are three hypotheses that might account for this behavior. First, the selective oxidation of chromium at the fretting surface might lead to an excessive buildup of oxidized debris in the 20-percent-chromium alloys at 650° and 816° C. At high temperatures, alloy softening leads to breakup of this oxide layer in the fretting region and damage to the alloy by the copious amounts of abrasive oxide particles. Second, the 20-percent-chromium alloys may have undergone more extensive fretting fatigue damage due to the diffusion of chromium to fatigue crack sites initiated in the fretting region, and the consequent acceleration of crack growth.

Third, the formation of a protective aluminum-rich oxide layer may be inhibited in the 20-percent-chromium alloys, but may be possible in the case of the 10-percent-chromium alloys.

Figures 5 and 6 show SEM photographs of the fretting wear scars of nickel - 10 percent chromium - 5 percent aluminum and of nickel - 20 percent chromium - 5 percent aluminum, respectively, after fretting at 650° C.

The most significant difference between the two wear scars is the presence of more spall-like pits over a large proportion of the wear scar area of the nickel - 20-percent-chromium - 5-percent-aluminum specimen. The spall-like pits are indicative of a fatigue mechanism. Also, the wear scar area surrounding the pits is more disrupted in the 20-percent-chromium - 5-percent-aluminum specimen. These observations support the second hypothesis, indicating that the higher chromium concentrations may accelerate surface fatigue damage.

The effectiveness of the higher aluminum concentration in reducing fretting damage can be related to some observations concerning the surface oxide formation in the wear scar. Elemental X-ray dispersion analysis of the nickel - 20-percent-chromium - 5-percent-aluminum and the nickel - 10-percent-chromium - 5-percent-aluminum alloy surfaces showed a significant difference in aluminum concentration after fretting at 650° C. Comparison of figures 7(a) and 8(a) indicates a proportionally much stronger aluminum peak in the fretting scar for the 10-percent-chromium - 5-percent-aluminum alloy than for the 20-percent-chromium - 5-percent-aluminum alloy. Also, as may be seen in figures 7(a) and (b), the aluminum peak is proportionally much higher within the fretting scar than outside it for the 10-percent-chromium - 5-percent-aluminum alloy (note the recycled nickel peak in fig. 7 (b)). Some comments concerning the significance of these X-ray dispersion data are in order here.

X-ray dispersion analysis is not strictly a surface examination technique. The peak heights result from surface region concentrations superimposed on subsurface bulk concentrations. Most likely, the only differences between material examined within and outside the fretting scar are in the surface region compositions. Therefore, it is justified to say that the difference in aluminum peak height within and outside the fretting scar for the 10-percent-chromium - 5-percent-aluminum alloy must reflect a real change in aluminum concentration on the fretting wear scar surface. No significant difference between the aluminum concentrations within and outside the fretting scar was observed on the 20-percent-chromium - 5-percent-aluminum alloy.

In an effort to gather further information on surface and near subsurface composition, the 20-percent-chromium - 5-percent-aluminum alloy was sectioned after fretting at 816° C, and X-ray dispersion analyses were made at various depths below the surface. This was done by setting the electron beam of the SEM on stationary mode, thereby illuminating a point about  $200 \times 10^{-10}$  to  $400 \times 10^{-10}$  meter (200 to 400 Å) in diameter.



Points at different depths were examined by electronically shifting the beam. The data from this study are shown in figure 9, and a graphical interpretation of these data is in figure 10. This figure is a plot of the ratio of the peak height of a given specie (e. g., aluminum) to the sum of the peak heights of all three species. A correction factor is applied to all data points in order to bring the data into agreement with the bulk composition at the interior points.

Figure 10 shows that the maximum chromium concentration occurs near the surface and that it approaches bulk concentration with increasing depth. Although there is a more-than-bulk concentration of aluminum near the surface, the maximum aluminum concentration occurs at 0.2 to 0.3 micrometer below the surface. This pattern was observed within and outside the fretting region, with minor variations in the relative amounts of aluminum and chromium near the surface at various locations. Thus, the elemental composition of the oxide film that first formed on the surface was not very different from that which formed later in the fretting scar, as subsurface material was exposed and oxidized.

The composition profiles of figure 10 suggest that the surface oxide of the 20-percent-chromium - 5-percent-aluminum alloy is made up of a mixture of oxides, possibly a mixture of nickel chromate and nickel aluminate spinels ( $\text{NiCr}_2\text{O}_4$  and  $\text{NiAl}_2\text{O}_4$ , respectively) in a layered structure. The variations with location observed in the surface composition further suggest that the film is nonhomogeneous, containing discrete particles. Such an oxide film structure may be prone to mechanical disintegration under fretting conditions, with the consequences of accelerated wear, as observed at  $650^\circ\text{C}$ , or the formation of debris buildups, as observed at  $816^\circ\text{C}$ .

Considered together, the X-ray dispersion results and fretting wear measurements suggest that the formation of an aluminum oxide ( $\text{Al}_2\text{O}_3$ ) or  $\text{NiAl}_2\text{O}_4$  surface film contributes significantly to the inhibition of fretting wear. It is proposed that such a film was able to form at high temperatures on the fretting wear scar surface of the 10-percent-chromium - 5-percent-aluminum alloy but was prevented from forming on the 20-percent-chromium alloys.

The important feature of the film formation in the nickel - 10-percent-chromium - 5-percent-aluminum alloy is that the film is evidently self-repairing, and protective to the surface. It is this self-repairing feature that would explain the enhanced aluminum concentration observed in the fretting scar of this alloy after 288 000 fretting cycles at  $650^\circ\text{C}$ . The fretting-resistant feature of the surface oxide film of this alloy is shown by the comparatively low fretting wear volumes measured at  $650^\circ$  and  $816^\circ\text{C}$ .

## SUMMARY OF RESULTS

The following are the principal results of this investigation of the fretting of nickel-chromium-aluminum alloys:

1. Due to the formation of protective oxide films, the alloys showed less fretting wear at all temperatures than did the high-purity nickel.
2. Increasing the chromium concentration from 10 percent to 20 percent was effective in reducing fretting wear at temperatures up to 540° C. At higher temperatures, an increase in fretting damage was observed in the 20-percent-chromium alloys.
3. Increasing the aluminum concentration from 2 percent to 5 percent effectively reduced the fretting wear at temperatures to 816° C.
4. The best alloy examined in this investigation was the nickel - 10 percent chromium - 5 percent aluminum. Its superior behavior was believed to be due to the formation of a protective, self-repairing oxide film at elevated temperatures.

Lewis Research Center,  
National Aeronautics and Space Administration,  
and  
U.S. Army Air Mobility R&D Laboratory,  
Cleveland, Ohio, October 30, 1973,  
501-24.

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TABLE I. - AVERAGE FRETTING COEFFICIENTS OF FRICTION

Material composition, wt. %	Coefficient of friction at -			
	23° C	540° C	650° C	816° C
99.9 Percent nickel	0.1 (in dry air) 0.15 to 0.2 (in saturated air)	---	0.2	---
Ni-10Cr-2Al	<0.1	0.3	0.3	0.4
Ni-10Cr-5Al	0.1	0.3	0.3	0.3
Ni-20Cr-2Al	0.1 to 0.15	0.2	0.15	0.25
Ni-20Cr-5Al	0.1	0.4	0.25 to 0.3	0.35 to 0.4

TABLE II. - FRETTING WEAR VOLUMES MEASURED ON FLAT SPECIMENS OF NICKEL AND NICKEL-CHROMIUM-ALUMINUM ALLOYS AFTER FRETTING AT VARIOUS TEMPERATURES

Material composition		Fretting wear volume (mm <sup>3</sup> ) after fretting <sup>a</sup> at -			
wt. %	at. %	23° C	540° C	650° C	816° C
99.9 Percent nickel	99.9 Percent nickel	200×10 <sup>-5</sup> 130 130 <sup>b</sup> 37 <sup>c</sup> 57 <sup>c</sup> 52	-----	65×10 <sup>-5</sup> 70	-----
Ni-10Cr-2Al	Ni-10Cr-4Al	200×10 <sup>-5</sup> 160	17×10 <sup>-5</sup>	26×10 <sup>-5</sup> 11	29×10 <sup>-5</sup> 19
Ni-10Cr-5Al	Ni-10Cr-10Al	100×10 <sup>-5</sup> 85	7.2×10 <sup>-5</sup> 7.2	10×10 <sup>-5</sup> 6	8.3×10 <sup>-5</sup> 7.0
Ni-20Cr-2Al	Ni-21Cr-4Al	100×10 <sup>-5</sup> 100	13×10 <sup>-5</sup>	34×10 <sup>-5</sup> 23	10×10 <sup>-3</sup> -mm buildup, <sup>d</sup> 0.28-mm diam 15×10 <sup>-3</sup> -mm buildup, <sup>d</sup> 0.30-mm diam
Ni-20Cr-5Al	Ni-21Cr-10Al	70×10 <sup>-5</sup>	4.2×10 <sup>-5</sup> 5.1	20×10 <sup>-5</sup> 9	3.0×10 <sup>-3</sup> -mm buildup, <sup>d</sup> 0.33-mm diam 4.0×10 <sup>-3</sup> -mm buildup, <sup>d</sup> 0.23-mm diam

<sup>a</sup>Specimens fretted in dry air unless noted otherwise.<sup>b</sup>Fretted in air with relative humidity of 25 percent.<sup>c</sup>Fretted in saturated air.<sup>d</sup>Wear volumes were not determined for specimens showing material buildup.

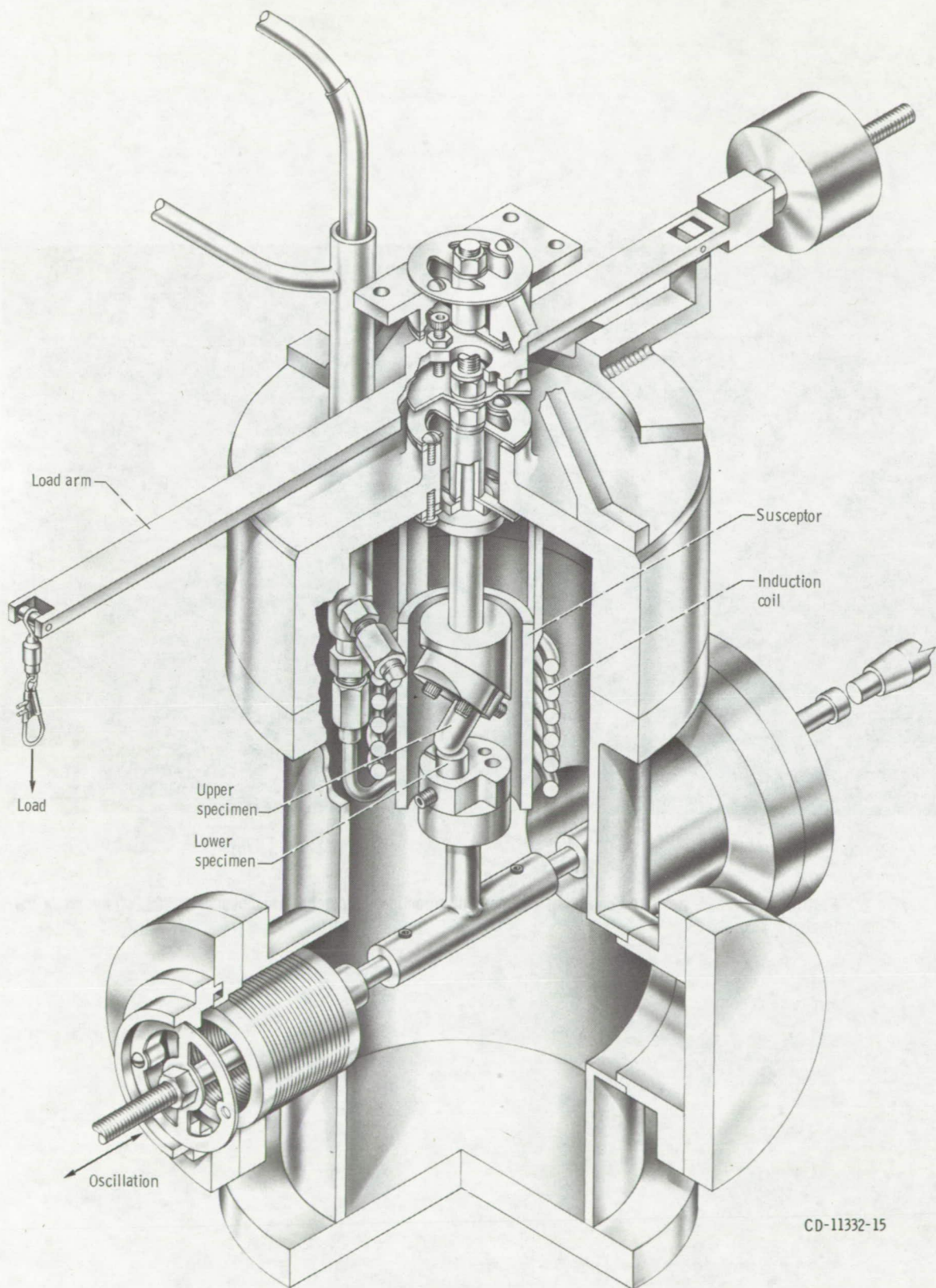
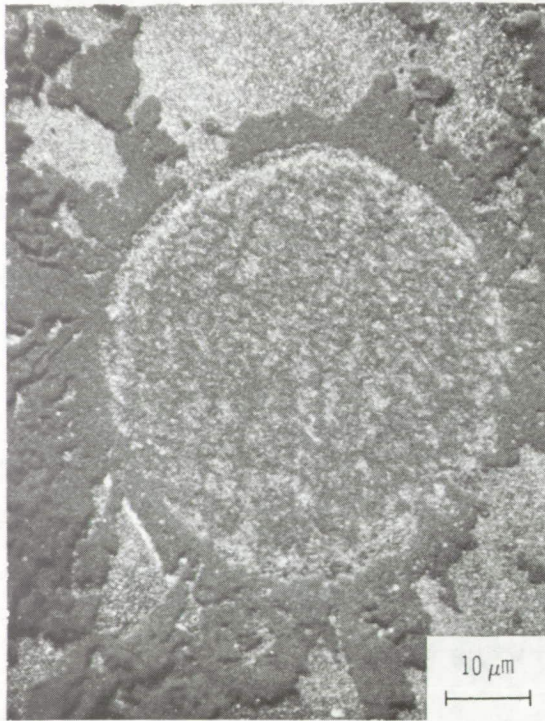


Figure 1. - Fretting apparatus.



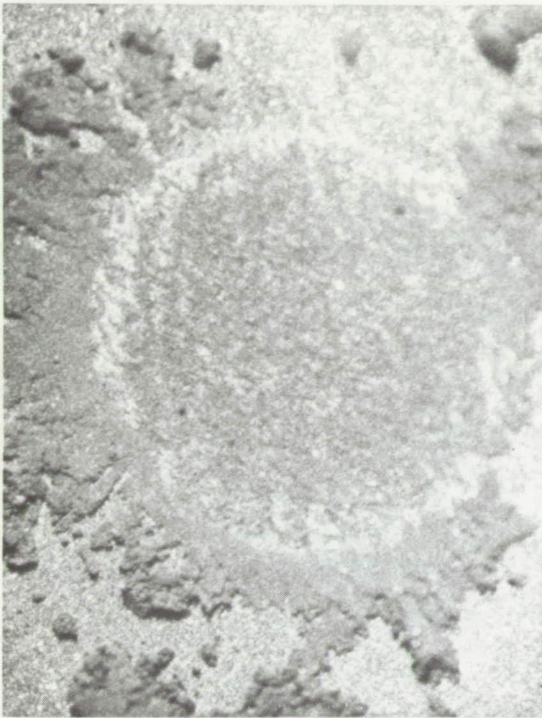


(a) Room temperature.

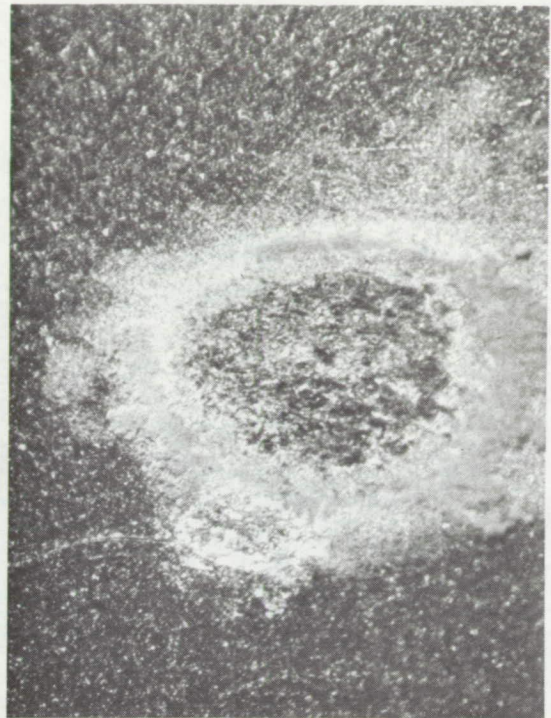


(b) 816°C.

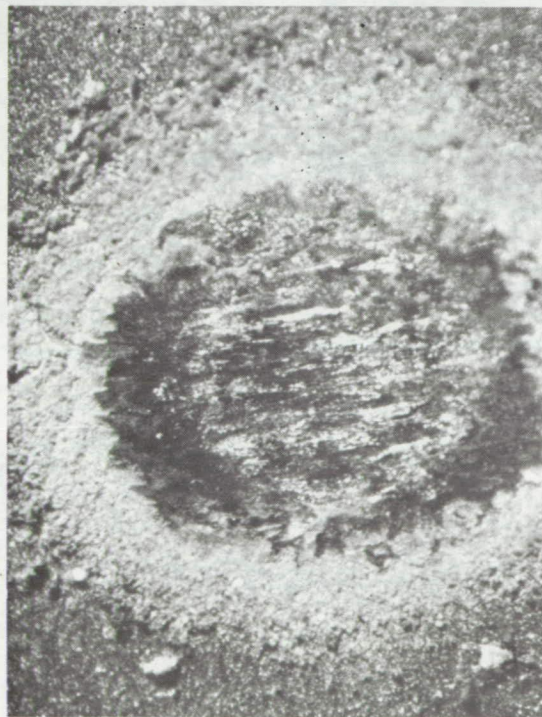
Figure 2. - Fretting wear scars produced on nickel - 20 percent chromium - 5 percent aluminum after 288 000 cycles (1 hr) at the indicated temperatures.



(a) Dry air.



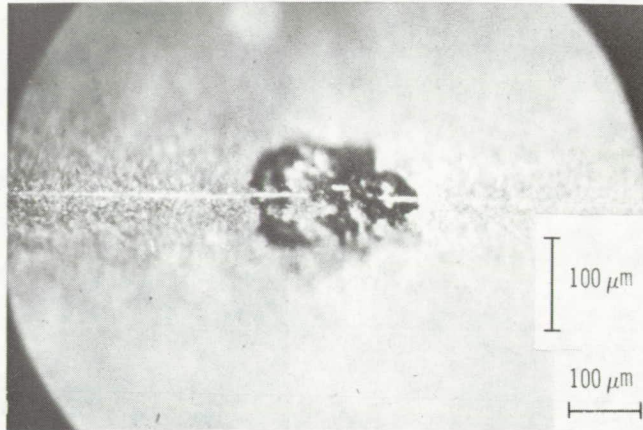
(b) Relative humidity of 25 percent.



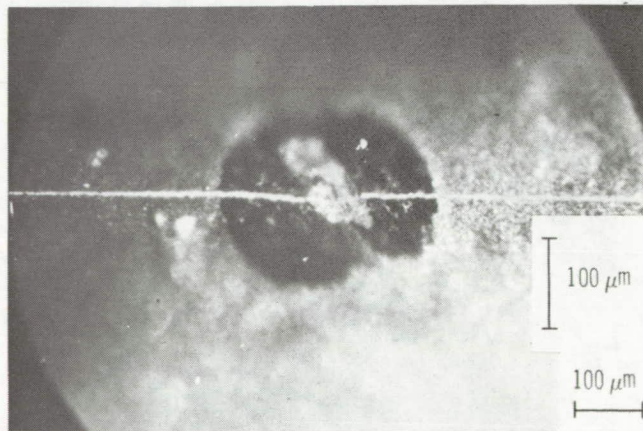
(c) Saturated air.

Figure 3. - Fretting scars produced on 99.9-percent-pure nickel at room temperature under various humidity conditions. X75.





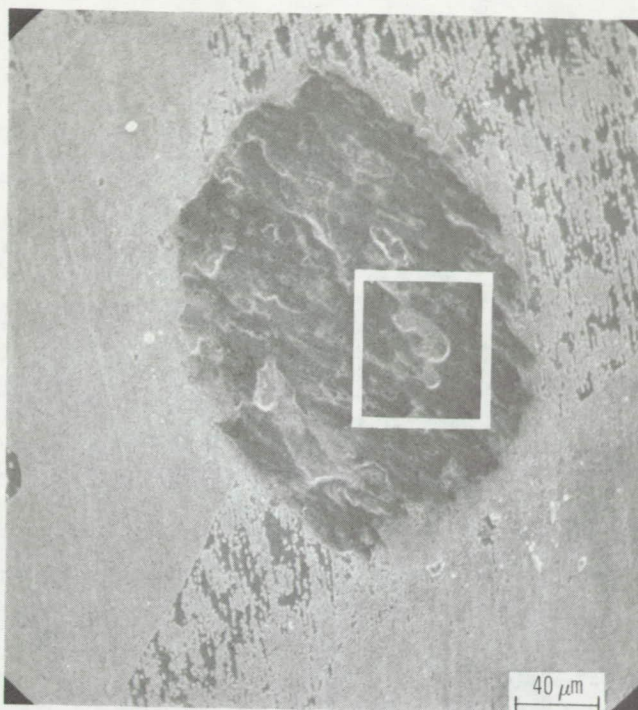
(a) Nickel - 20 percent chromium - 2 percent aluminum.



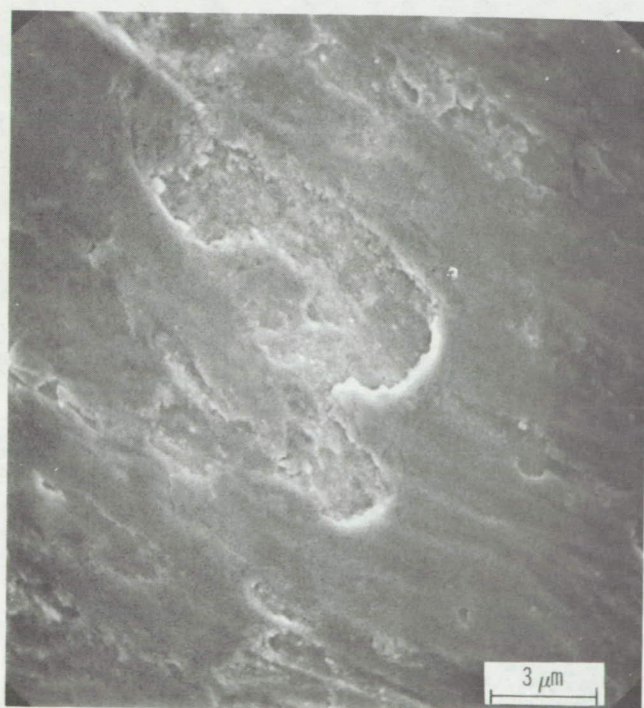
(b) Nickel - 10 percent chromium - 2 percent aluminum.

Figure 4. - Light section micrographs showing the surface profiles of two nickel-chromium-aluminum alloys after fretting in dry air at 816° C (1500° F).



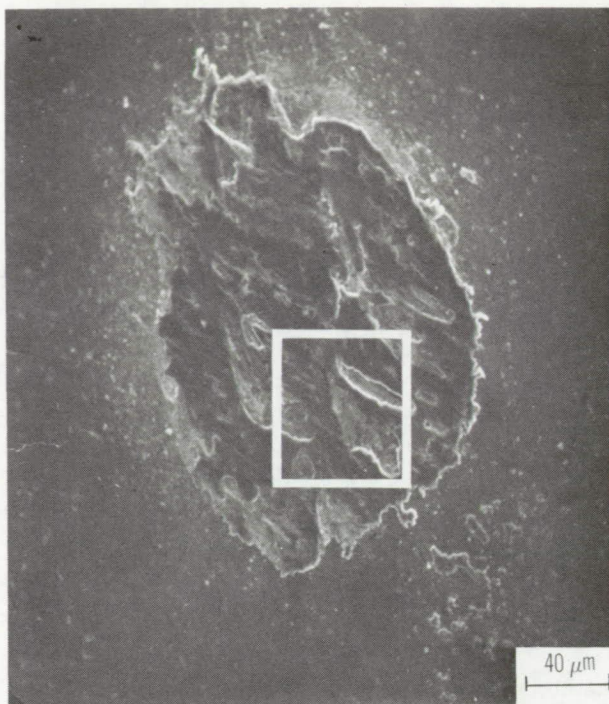


(a) Overview.

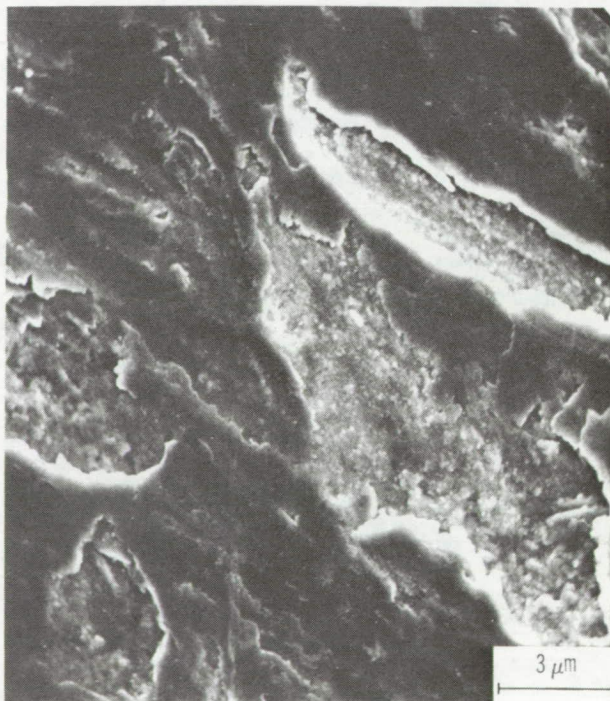


(b) Boxed area of part (a).

Figure 5. - SEM photographs of nickel - 10-percent-chromium - 5-percent-aluminum alloy fretted in dry air at 650°C.



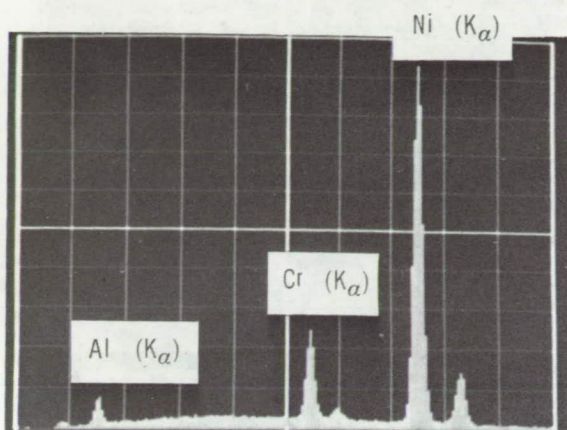
(a) Overview.



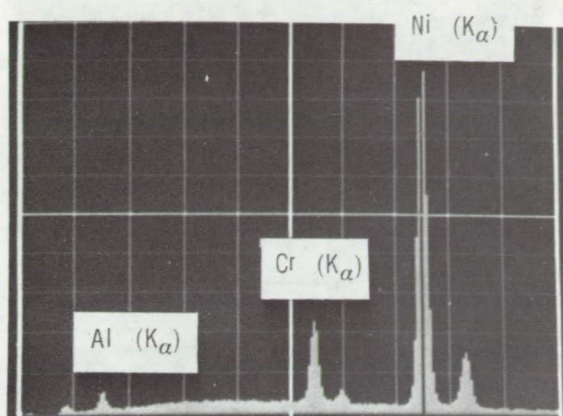
(b) Boxed area of part (a).

Figure 6. - SEM photographs of nickel - 20-percent-chromium - 5-percent-aluminum alloy fretted in dry air at 650°C.



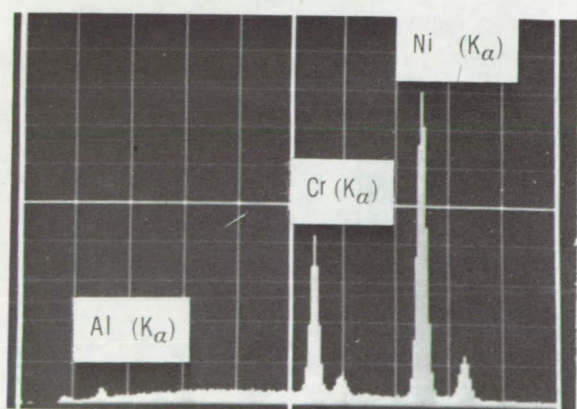


(a) Within fretting scar.

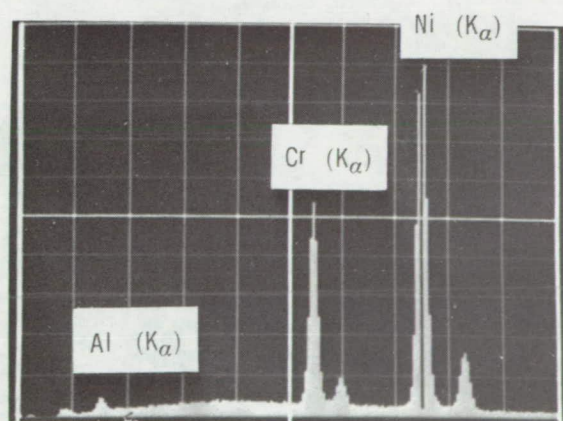


(b) Outside fretting scar.

Figure 7. - X-ray dispersion analyses of nickel - 10-percent-chromium - 5-percent-aluminum alloy within and outside the fretting scar after fretting in dry air at 650° C.

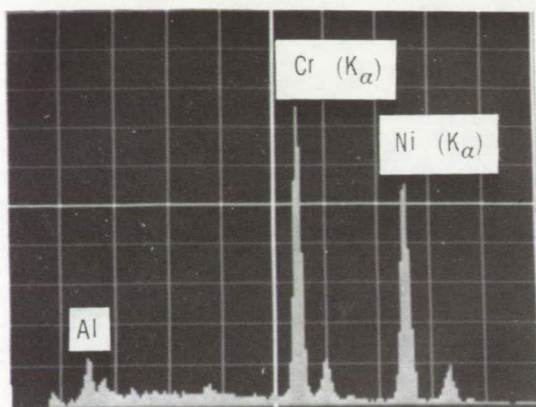


(a) Within fretting scar.

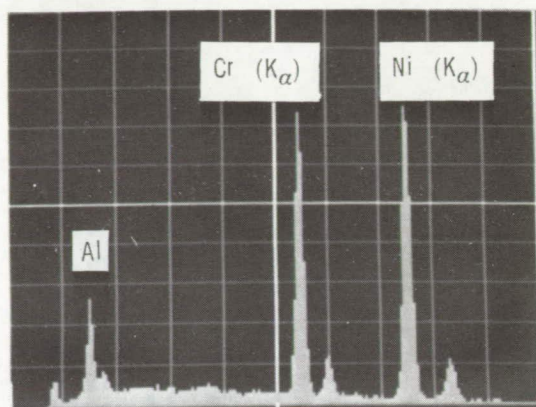


(b) Outside fretting scar.

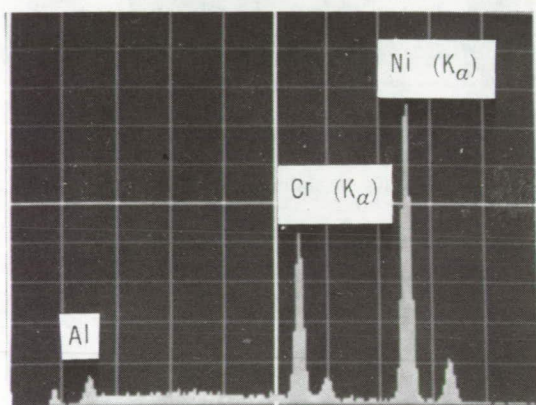
Figure 8. - X-ray dispersion analyses of nickel - 20-percent-chromium - 5-percent-aluminum alloy within and outside the fretting scar after fretting in dry air at 650° C.



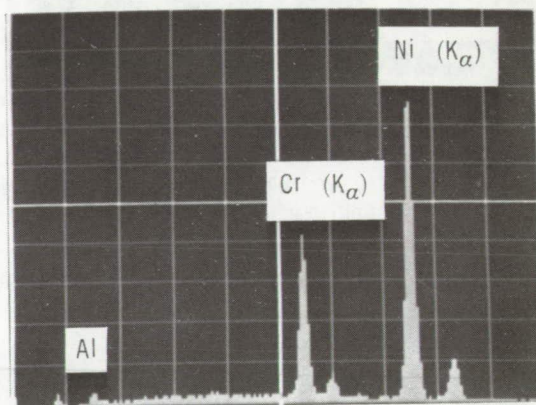
(a) Near surface.



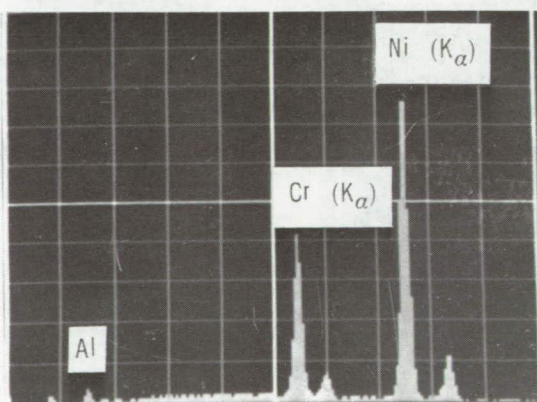
(b) At depth of 0.25 micrometer.



(c) At depth of 1 micrometer.



(d) At depth of 4 micrometers.



(e) Near center of specimen.

Figure 9. - Elemental X-ray dispersion analyses at various depths below the surface of a sectioned nickel - 20-percent-chromium - 5-percent-aluminum specimen after 288 000 fretting cycles (1 hr) at 816° C (1500° F).

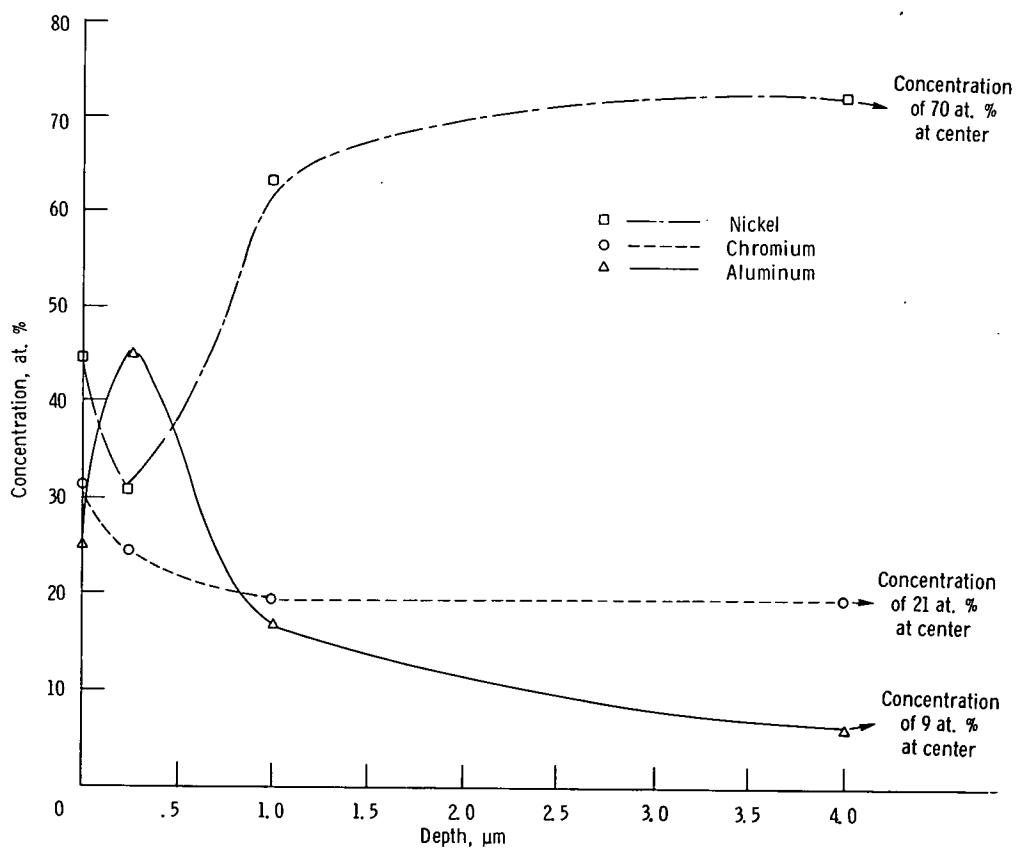


Figure 10. - Composition as a function of depth for nickel - 20-percent-chromium - 5-percent-aluminum alloy after 288 000 fretting cycles (1 hr) at 816° C (1500° F).





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